

#### PATENT APPLICATION

#### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

#### DECLARATION UNDER 37 C.F.R. §1.132

We, Naoki Sasaki and Mari Yoshida, both citizens of Japan, both hereby declare and state:

- I, Naoki Sasaki, graduated from the Yamagata University in March 1999, and started working at Kokyu Alcohol Kogyo Co., Ltd in April, 1999; and, since then, have been engaged in research on polymer synthesis.
- 2. I, Mari Yoshida, graduated from the Kochi University of Technology in March 2004, and started working at Kokyu Alcohol Kogyo Co., Ltd in April 2004; and, since then, have been engaged in research and development.
- 3. We are aware of and familiar with U.S. Patent No. 6,242,499 B1 (hereinafter "Gruning"). We are also familiar with the specification and claims of U.S. Patent Application Serial No. 10/590,151 (hereinafter the "151 application") and No. 11/371,290 (hereinafter the "290 application").
- 4. We have a professional relationship with Kokyu Alcohol Kogyo Co., Ltd. In the course of the professional relationship, we received compensation directly from the assignee for our work relating to polymer research and development. We are being compensated for our work in connection with this Declaration.
- 5. We and/or those under our direct supervision and control have conducted the following tests from the 5<sup>th</sup> to the 8<sup>th</sup> of April 2010, as described in detail below.

#### I. Introduction

The following three compositions were tested: (1) Lot 100407; (2) HAILUCENT; and (3) ISOLAN® PDL Lot 100407 was selected and prepared as a Comparative Example representative of Gruning. HAILUCENT was prepared as an example according to the specification of the '151 application, and ISOLAN® PDI is a commercial example indicative of Gruning.

The synthesis and preparation of Lot 100407 is described below, and each of the three experimental compositions is summarized in Table 2 as follows.

#### A. Synthesis of Lot 100407

A polyglycerol ester was prepared in a manner identical to the EXAMPLES of Gruning (see Gruning, col. 4).

#### 1) Materials

The materials used in the preparation of Lot 100407 are listed below in Table 1:

Table 1
Materials for Preparation of Lot 100407

Materials for Preparation of Lot 100407					
Isostearic acid	Prisoline 3507, ex. Unichema				
Polyglycerol	Polyglycedrol-3, ex. Solvay Chemicals GmbH, having the following oligomer distribution, as certified by Solvay (se attached certificate from Solvay Chemicals GmbH):				
784	Diglycerol	27.9 %			
	<ul> <li>Triglycerol</li> </ul>	46.0 %			
	<ul> <li>Tetraglycerol</li> </ul>	17.9 %			
	<ul> <li>Pentaglycerol</li> </ul>	5.6 %			
	Hexaglycerol and higher	2.6%			
Dimer acid	Pripol 1025, ex. Unichema, with an aver polymerization of 2.4, as used in Grunin line 34)				

#### 2) Preparation of Lot 100407

264 g of isostearic acid and 100 g of polyglycerol were added to 100 g of methycyclohexane, and heated to reflux under a nitrogen flow while separating water formed from the reaction. After the temperature of the reaction mixture reached 250°C, the temperature was maintained for 3 hours, which marked the end point of the first reaction step.

The reaction mixture was cooled to a temperature of 80°C. Subsequently, 121 g of dimer acid was added to the reaction mixture, and the mixture was heated to reflux under a mitrogen flow while separating water formed. After the temperature reached 250°C, the temperature was maintained for 3 hours, which marked the end point of the second reaction step.

The reaction mixture was cooled to 150°C. Subsequently, the pressure was decreased to recover methycyclohexane, while paying attention to avoid solvent bumping. After maintaining a temperature of 150°C at a pressure of approximately 7 mm Hg/cm² for 2 hours, the recovery of the solvent was completed. The reaction mixture was allowed to cool to room temperature, and the vacuum and nitrogen flow were stopped. The finished product is heretofore referred to as "Lot 100407."

#### B. Experimental Compositions

Table 2 on the following page summarizes the three experimental compositions that were tested.

Table 2

Experimental Compositions				
COMPOSITION DESCRIPTION		DESCRIPTION		
1	Lot 100407  Comparative Example of  Gruning	See section above for synthesis of Lot 100407		
2	HAILUCENT  Example according to Preparation Example 3 of the Specification	HAILUCENT is a product of Kokyu Alcohol Kogyo Co., Ltd  HAILUCENT was prepared in a manner identical to Preparation Example 3 of the present specification, except that the diglycerol used was "Diglycerin 801" ex. Sakamoto Yakuhin Kogyo Co., Ltd., instead of K COL II (see specification, paragraphs [0035] and [0036]).  Diglycerin 801 is equivalent to K COL II and was used because K COL II is no longer being produced (see specification, paragraph [0023]).  The isostearic acid, "isostearic acid EX", used in "HAILUCENT" is a purified one from Prisoline 3507, ex. Unichema; and the dimer acid, "PRIPOL1009", used in "HAILUCENT, has an average degree of polymerization of 2.0 (almost pure).  The mole ratio of diglycerin: isostearic acid: dimer acid in "HAILUCENT" is 1: 1.5: 0.65.		
3	ISOLAN® PDI  Comparative  Commercial Example of  Gruning	ISOLAN® PDI is a product of Goldschmidt AG, the assignce of Gruning (U.S. Patent No. 6,242,499).  The term "ISOLAN® PD1" is the trade name for the INCI/CTFA name: diisostearoyl polyglyceryl-3 dimer dilinoleate.   Include the state of Goldschmidt AG, the assignce of Gruning (U.S. Patent No. 6,242,499).		

<sup>&</sup>lt;sup>1</sup> see, e.g. http://www.manufacturingchemist.com/company/single\_company/Evonik\_Goldschmidt\_GmbH/46023 (last visited: 15 May 2010).

#### II. Methodology and Analysis

#### A. Number Average Molecular Weight and Viscosity

The number average molecular weight and viscosity were measured for the three experimental compositions listed in Table 2. The number average molecular weight was measured by gel permeation chromatography (GPC), and the viscosity for each composition was measured at 60°C.

#### B. Removal with Water and Gloss

An evaluation for removal with water, and an evaluation for gloss properties were conducted for the three experimental compositions listed in Table 2.

The evaluation for removal with water was conducted as follows:

Five panelists participated in the evaluation. Approximately one gram of a sample was applied to an area of about 30 mm x 30 mm on a skin of a lower inside arm of the panelists. The sample was left for 30 minutes. After 30 minutes passed, the skin area was washed with soap, while paying attention to prevent fingers from coming into direct contact with the applied skin area. Then, the applied skin area was rinsed with running water for one minute. The panelists were asked to evaluate a degree that they felt of any remaining substance on the skin using five grades, ranked in order of degree from "strong remain" to "no remain".

The evaluation for gloss was conducted as follows:

Approximately one gram of a sample was applied on paraffin paper in an area of 20 mm x 40 mm and a thickness of about 200 microns. Gloss was measured with Gross Checker IG-330, ex. Horiba Seisakusyo, at an incident angle of 60 degrees and a reflection angle of 60 degrees. Measurements for each sample were conducted three times. The results were then averaged.

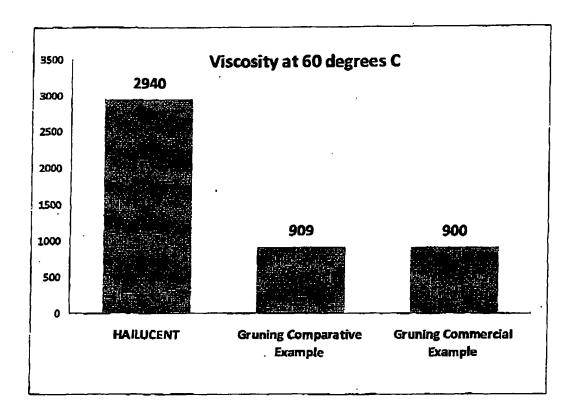
The results and measurements of the number average molecular weight, the viscosity at 60°C, removal with water test and gloss test are summarized below in Tables 3, 4 and the accompanying Graph:

Table 3
Number Average Molecular Weight, Viscosity, Removal with Water and Gloss

Experimental Composition	Number average molecular weight	Viscosity at 60°C; [mPa_s]	Removal with Water	Gloss
HAILUCENT  Example According  Preparation  Example 3 of the  Specification	4936	2940	"strong remain" by all of the five panelists	79
Lot 100407  Comparative  Example of Gruning	2927	909	"no remain" by all of the five panelists	70
ISOLAN® PDI  Comparative  Commercial  Example of Gruning	2856	900	"no remain" by all of the five panelists	68

Table 4
Percent Difference

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	Viscosity at 60°C [mPa.s]	Gloss		
HAILUCENT vs. Lot 100407	223.4% increase	12.8% increase		
	over Lot 100407	over Lot 100407		
HAILUCENT vs. ISOLAN® PDI	226.7% increase	16.1% increase		
	over ISOLAN® PDI	over ISOLAN® PDI		



As illustrated by the above results, the composition of the present specification (HAILUCENT) has a viscosity at 60°C, removal with water and gloss that are significantly higher than those of both the Comparative Prepared Example of Gruning (Lot 100407) and the Comparative Commercial Example of Gruning (ISOLAN® PDI).

The sizeable difference with respect to viscosity and gloss; and the significant improvement in removal with water properties that was created as a result of preparing a product with a diglycerol as recited in claim 1 of the '151 application, was unexpected from similar comparative products that were prepared with a mixture of polyglycerols absent from the components recited in claim 1 of the '151 application.

We hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so

made are punishable by fine and/or imprisonment under Section 1001 of Title 18 of the
United States Code, and that such willful false statements may jeopardize the validity of the
application or any patent issuing therefrom.

Date: July, 30, 2010

Nachi Secali

Date: July, 30, 2010

Mari Yoshida Mari Yoshida

Attachment:

Certificate from Solvay Chemicals GmbH



# SOLVAY CHEMICALS GmbH

### **CERTIFICATE OF ANALYSIS**

norm EN 10204 - 3.1 B PDS-2820-0007

Batch/Lot/Charge No. : RBA081008A

Ludwigstr. 10 47495 Rheinberg Telefon 02843 73 2571 Telefax 02843 73 3746

Customer

: NIPPON SOLVAY k.k

Toranomon 30 Mori Building, Toranomon 3-2-2, Minatoku-Tokyo,105-0001, Japan Attention of: Kitsumoto Kondo

Date of our delivery Quantity of our delivery :04.11.2009

:5 x 1kg

Delivery in

:Bottle

## POLYGLYCEROL-3

#### Test results

Parameter	Concentration- unit	Analytical - result	Specification
Diglycerol	%	27,9	15 - 30
Triglycerol	%	46,0	35 - 55
Tetraglycerol	%	17,9	10 - 25
Pentaglycerol	%	5,6	<= 10
Hexaglycerol and higher	%	2,6	< <b>=</b> 5
Color	APHA	45	< 250
Water	g/kg	0,3	<= 1
Chloride	ma/ka	0,2	< 100

We hereby certify, that the material described above has been tested and complies with the terms of the order contract. Results displayed as "conform" are certified under EN 10204 2.1 standard (certificate of conformity).

Abt.: L-QO

Rheinberg,

04.11.2009

gez. B.Brünken

Works Inspektor

Some applications of this product may be regulated or restricted by national or international standards (e.g. for food additives, water treatment, the pharmaceutical industry, etc). The buyer and the eventual user, in his sole and entire liability, shall respect those standards, orders of any relevant authority, and all existing patents and intellectual properties rights; and shall comply with the laws and the regulations applicable to our products and/or to his activity. The buyer and the eventual user must independently determine the suitability of this product for any particular purpose and its manner of use.

Please contact us for further information on grades developed for a specific end-use.

**10.3.24** 前集高建制